

**Quality Assurance Project Plan  
Title and Approval Sheet  
Final Version, October 4, 2001  
Raisin River Sediment Sampling in FY2002:  
A Follow-Up to the 1997 Sediment Remediation Project  
IAG#DW96947964-01**

---

Scott Cieniawski, Project Officer, GLNPO  
77 West Jackson Blvd (G-17J), Chicago, Illinois 60604-3590, Office Phone:312-353-9184,  
Cellular Phone (field) 312-961-0592, Fax: 312-353-2018

---

Paul Baxter, Project Coordinator, U.S. Army Corps of Engineers - Detroit District  
19215 W. Eight Mile Road, Detroit, Michigan 48226,  
Phone (313) 226-7555, Fax (313) 226-2013

---

Patricia Novak, Project Manager, Lakeshore Engineering Services, Inc.  
19215 W. Eight Mile Road, Detroit, Michigan 48219,  
Phone (313) 535-7882, Fax (313) 535-7875

---

Ann Preston, Project Manager, Trace Analytical Laboratories, Inc.  
2241 Black Creek Rd., Muskegon, Michigan 49444-2673,  
Phone (231) 773-5998, Ex. 224, Fax (231) 773-6537

---

Al Mozol, Operations Manager, ASci Corporation  
444 Airpark Blvd., Duluth, Minnesota 55811,  
Phone (218) 722-4040, Fax (218) 722-2592

---

Kathleen Loewen, Quality Assurance Manager, Lancaster Laboratories  
2425 New Holland Pike, Lancaster, Pennsylvania 17605-2425,  
Phone (717) 656-2300, Fax (717) 656-0450

---

Louis Blume, Quality Assurance Manager, GLNPO  
77 W. Jackson Blvd. (G-17J), Chicago, IL 60604  
Phone (312) 353-2317, Fax (312) 353-2018

## QAPP Distribution List

Scott Cieniawski, Project Officer  
Lou Blume, QA Manager  
USEPA- GLNPO  
77 West Jackson Blvd (G-17J)  
Chicago, Illinois 60604-3590  
phone: 312-353-9184

Paul Baxter, Project Coordinator  
U.S. Army Corps of Engineers - Detroit District  
477 Michigan Avenue  
Detroit, MI 48226  
phone: 313-226-7555

Roger Jones  
Michigan DEQ - SWQD  
P.O Box 30273  
Lansing, MI 48909  
phone: 517 373-4704

Patricia Novak, Project Manager  
Lakeshore Engineering Services, Inc.  
19215 W. Eight Mile Road, Detroit, Michigan 48219  
phone: (313) 535-7882

Ann Preston, Project Manager  
Trace Analytical Laboratories, Inc.  
2241 Black Creek Rd., Muskegon, Michigan 49444-2673  
phone: (231) 773-5998, Ex. 224

Al Mozol, Operations Manager  
ASCI Corporation  
4444 Airpark Blvd., Duluth, Minnesota 55811  
phone: (218) 722-4040

Kathleen Loewen, Quality Assurance Manager  
Lancaster Laboratories  
2425 New Holland Pike, Lancaster, Pennsylvania 17605-2425  
phone: (717) 656-2300

**TABLE OF CONTENTS**

QAPP Signature Page . . . . .	1
QAPP Distribution List . . . . .	2
Table of Contents . . . . .	3
1. Summary . . . . .	5
1.1 Purpose . . . . .	5
1.2 Background . . . . .	5
1.3 Project Organization . . . . .	5
2. Project Description . . . . .	10
2.1 Data Uses and Expected Measurements. . . . .	10
2.2 Criteria and Objectives . . . . .	12
2.2.1 Sediment Chemistry . . . . .	12
2.2.2 Fish Tissue and <i>L. variegatus</i> Tissue Chemistry . . . . .	13
2.3 Special Personnel, Training, and Equipment Requirements . . . . .	13
2.4 Project Schedule . . . . .	14
3. Sampling Plan . . . . .	14
3.1 Sampling Network Design and Rationale . . . . .	14
3.2 Definition of Sample Types . . . . .	16
3.3 Type and Number of Samples . . . . .	17
3.4 Field Data Collection . . . . .	17
4. Sample Collection and Handling. . . . .	18
4.1 Sample Collection. . . . .	18
4.1.1 Sediment Cores . . . . .	18
4.1.2 Sediment Ponars . . . . .	18
4.1.3 Cage Fish Samples . . . . .	19
4.2 Sample Handling . . . . .	19
4.2.1 Sample Containers . . . . .	19
4.2.2 Sample Labeling . . . . .	20
4.2.3 Shipping and Chain-of-Custody . . . . .	21
4.2.4 Receipt of Samples . . . . .	22
5. Laboratory Analysis . . . . .	23
5.1 Analysis Methods . . . . .	23
5.2 Data Quality Objectives. . . . .	24
5.2.1 Method Detection Limits and Level of Quantitation . . . . .	24
5.2.2 Bias . . . . .	26
5.2.3 Precision . . . . .	26
5.2.4 Accuracy . . . . .	28
5.2.5 Representativeness . . . . .	28
5.2.6 Comparability . . . . .	28
5.2.7 Completeness . . . . .	29
6. Documentation and Records . . . . .	29
6.1 Field Documentation . . . . .	29
6.2 Laboratory Reports . . . . .	30
7. Special Training Requirements . . . . .	31
8. Quality Control Requirements . . . . .	31
8.1 Instrument/Equipment Testing, Inspection, and Maintenance Requirements . . . . .	32
8.2 Instrument Calibration and Frequency . . . . .	32
8.3 Inspection/Acceptance Requirements for Supplies and Consumables . . . . .	33
8.4 Data Management . . . . .	34
8.5 Data Acquisition Requirements (Non-Direct) . . . . .	36

**Table of Contents**

9.	Assessment and Oversight . . . . .	36
9.1	Assessment and Response Actions . . . . .	36
9.2	Reports to Management . . . . .	39
10.	Data Validation and Usability . . . . .	40
11.	References . . . . .	41

**List of Figures**

<b>Figure 1.</b>	Organizational Chart . . . . .	7
<b>Figure 2.</b>	Sampling Locations . . . . .	15
<b>Figure 3.</b>	Example Sample Label . . . . .	21

**List of Tables**

<b>Table 1.</b>	Screening Values for Contaminants of Concern . . . . .	13
<b>Table 2.</b>	Required Reporting Limits for Tissue Chemistry to Allow Comparison to Historical and Future Data Sets . . . . .	13
<b>Table 3.</b>	Tentative Project Schedule . . . . .	14
<b>Table 4.</b>	Summary of Data and Analyses at Sampling Locations . . . . .	16
<b>Table 5.</b>	Summary of Type and Number of Samples to be Collected . . . . .	17
<b>Table 6.</b>	Sample Container and Preservation Requirements. . . . .	20
<b>Table 7.</b>	Addresses for Shipment of Samples . . . . .	22
<b>Table 8.</b>	Laboratory Analysis and Preparation Methods. . . . .	23
<b>Table 9.</b>	Target Detection Limits for Sediment Chemistry . . . . .	25
<b>Table 10.</b>	Target Detection Limits for Tissue Chemistry . . . . .	25
<b>Table 11.</b>	Quality Control Limits . . . . .	27

**Appendices**

Appendix A:	Sampling SOPs
Appendix B:	Benthic Community Assessment SOP
Appendix C:	Caged Fish Sampling SOP (GLEAS Procedures #62 and #31)
Appendix D:	Field Measurement SOP
Appendix E:	Field Sample Log
Appendix F:	USACE Survey Markers
Appendix G:	Example Chain of Custody Form
Appendix H:	Cooler Receipt Form
Appendix I:	Laboratory Method Detection Limits, Method Reporting Limits, & SOP Numbers
Appendix J:	Minimum QA/QC Checklist for Post-Sampling Data Evaluation
Appendix K:	Summary Tables for Sample Collection and Analysis

## 1. SUMMARY

### 1.1 Purpose

The sampling efforts detailed in this document outlines a plan to determine the extent of recovery of the aquatic system at the site of the 1997 River Raisin - Ford Outfall Site sediment remediation project. The data collected during this study will be used to compare with data collected prior to the remediation project. The data will also be used to compare with future data to document the progress of the system's recovery over time. Proposed testing for this round of sampling include:

1. Caged Fish Testing
2. Sediment Bioaccumulation Testing (with *Lumbriculus variegatus*),
3. Sediment Chemistry (PCBs, metals, SVOCs, SEM-AVS, TOC, nutrients)
4. Whole Sediment Toxicity Testing (with *Hyaella azteca* & *Chironomus tentans*),
5. Benthic Community Analysis (specific to lowest taxonomic level below family)

Primary Objective: Collect sufficient data to ascertain the current state of the sediment environment in the vicinity of the Raisin River, Ford Outfall, Sediment Removal Project

Secondary Objectives: 1. Collect sufficient data to compare current sediment chemistry and caged fish testing data to historical data collected at the site.

2. Collect adequate sediment data (chemistry, toxicity, tissue chemistry, etc.) to provide an overall summary of sediment quality conditions within the Raisin River AOC.

### 1.2 Background

#### Site Location

The Raisin River flows southeast through the southeast corner of Michigan's Lower Peninsula, discharging into Lake Erie at Monroe Harbor near the city of Monroe, Michigan. The Area of Concern (AOC) is defined as "the lower 2.6 miles of the River Raisin, downstream from Dam Number 6 at Winchester Bridge in the city of Monroe, extending [downstream] one-half mile into Lake Erie, and including Plum Creek which discharges into Lake Erie through a canal." (USEPA, 1994)

Once forested with mature hardwood stands, the AOC now consists of mostly cleared land that is urban, suburban, and industrial in nature. Industries within the AOC include automotive, steel and paper manufacturers. Several landfills also border the river within the AOC. Contaminants of concern in the AOC include: polychlorinated bi-phenols (PCBs), chromium, copper, zinc, and oil and grease. (USEPA, 1994).

#### Remedial Activities

In 1997 the U.S. Environmental Protection Agency (USEPA) completed a Superfund removal project at the Ford Monroe contaminated sediment site. The cleanup resulted in the remediation of approximately 27,000 cubic yards of PCB-contaminated sediments (maximum concentration 49,000 ppm) by the Ford Motor Company from a large depositional area near an old 48-inch outfall from the Ford Monroe Stamping Plant. Remedial work also included the removal of

contaminated in-plant sewer material to eliminate the potential for on-going releases. The remedial work at the site was completed in October 1997. (USEPA, 1998).

### Historical Sampling

A significant amount of sediment chemistry and caged fish testing data is available to document pre-remedial conditions at the site. A limited amount of post-remediation data is also available. If available, the following historical data sets will be evaluated as part of this project:

1. 1988 Michigan DNR caged fish testing,
2. April 1991 sediment sampling survey by Michigan State University,
3. November 1991 sediment sampling survey by Michigan DNR's Surface Water Quality Division,
4. 1991 Michigan DEQ caged fish testing,
5. October 1992 sediment sampling survey by Region 5 USEPA,
6. 1995 Michigan DEQ sediment sampling survey,
7. 1997 Michigan DEQ sediment sampling survey
8. 1998 Michigan DEQ caged fish testing,
9. 1998 Michigan DEQ sediment sampling survey, and
10. 2000 Army Corps of Engineers sediment sampling survey.

### Selection of Site for Post-Remediation Testing

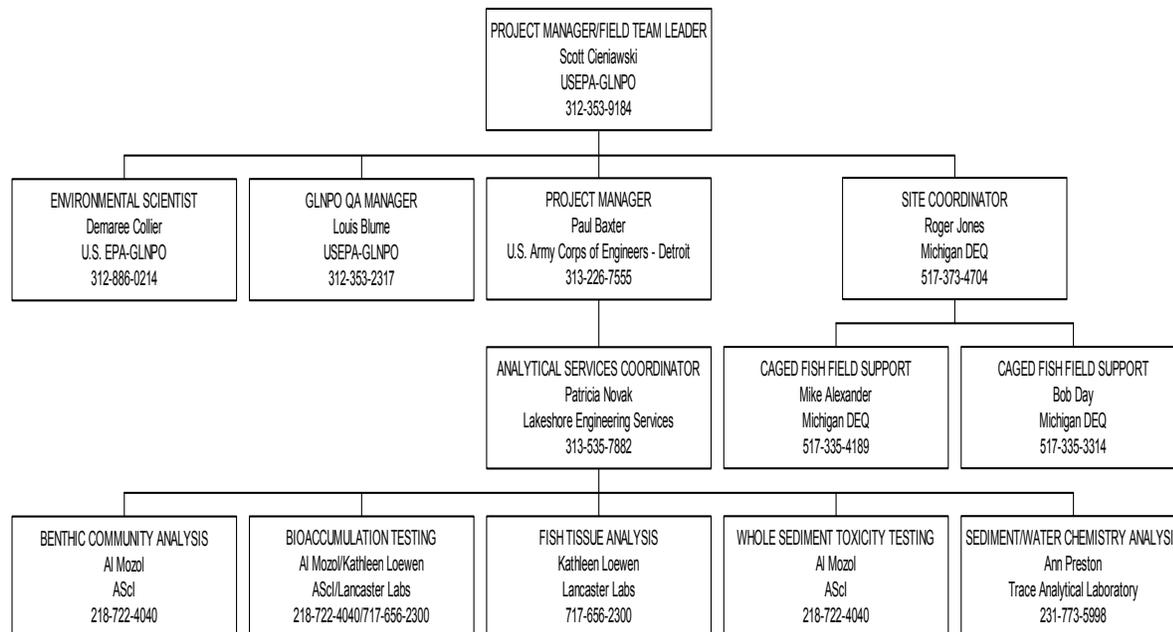
The Great Lakes National Program Office (GLNPO) has been interested in collecting data on: (1) the ability of environmental dredging projects to reach remedial targets, (2) levels of residual contamination left behind, and (3) recovery of the aquatic system after completion of environmental dredging projects. However, in order to address these questions, significant pre-remedial data must exist for a site, a major sediment remediation project must have been completed that addresses a significant portion of the contamination present at the site, and, based on information gained on post-remedial assessments at the Black River Ohio site, the aquatic system must be given a minimum of three years to recover before post-remedial work is performed.

GLNPO believes that the Raisin River is one of the few sites within the Great Lakes Basin that meets all three of these criteria. It has now been approximately four years since sediment remediation and source control work was completed at the Ford Monroe sediment site. The Ford Monroe removal represents a significant portion of the PCB contamination present at the site, and significant pre-remedial sediment and caged fish data exists for the AOC.

## **1.3 Project Organization**

Table 1 provides a summary of the project organization for this project. A description of the duties of each individual is provided below.

**Figure 1. Organizational Chart**



**USEPA-GLNPO**

USEPA-GLNPO is the principal investigating agency for this sediment survey. They are responsible for coordination and development of the sampling plan and QAPP as well as the principal client for the final data. USEPA-GLNPO staff associated with this project include:

**Person:**

Scott Cieniawski  
Project Coordinator/Field Team Manager  
77 W. Jackson Blvd. (G-17J)  
Chicago, IL 60604  
phone: 312-353-9184  
cieniawski.scott@epa.gov

**Responsibilities:**

Prepare Sampling Plan  
Prepare QAPP  
Oversee Sample Collection  
Field Team Member  
Analyze data and write report  
Perform project management tasks

Demaree Collier  
Environmental Scientist  
77 W. Jackson Blvd. (G-17J)  
Chicago, IL 60604  
phone: 312-886-0214  
collier.demaree@epa.gov

Review/Analyze Data  
Field Team Member

Louis Blume  
GLNPO QA Manager  
77 W. Jackson Blvd. (G-17J)  
Chicago, IL 60604  
phone: 312-353-2317  
blume.louis@epa.gov

Review/Approve QAPP

U.S. Army Corps of Engineers - Detroit District/USACE HTRW Center of Expertise

The U.S. Army Corps of Engineers will provide laboratory contracting support, and an initial QA/QC review of the project data. The USACE representatives will be responsible for contacting USEPA-GLNPO regarding any concerns regarding the data received from the laboratories, and advising USEPA-GLNPO regarding any concerns expressed by the laboratories. USACE individuals involved in this project include:

***Person:***

Paul Baxter  
Project Coordinator  
313-226-7555  
Paul.R.Baxter@lre02.usace.army.mil

***Responsibilities:***

Contract out analytical work  
Perform contract management activities  
Review QAPP

Cheryl Groenjes  
Chemist  
402-697-2568  
Cheryl.A.Groenjes@nwd02.usace.army.mil

Perform QA/QC review of the analytical report

Michigan DEQ

The Michigan DEQ will provide coordination and field support to this project. Field support will be provided during sediment sampling and MDEQ will conduct the caged fish sampling. MDEQ will also provide historical data, results, and information on sampling and analysis methods used during historical studies.

***Person:***

Roger Jones  
Site Coordinator - MDEQ - SQWD  
P.O. Box 30273  
Lansing, MI 48909-7973  
517-373-4704  
jonesrjj@state.mi.us

***Responsibilities:***

Coordinate MDEQ Support to Project  
Provide information on historical sampling results and analytical methods  
Review final report  
Review QAPP

Mike Alexander  
P.O. Box 30273  
Lansing, MI 48909-7973  
alexandm@state.mi.us

Conduct Caged Fish Sampling  
Provide SOP for caged fish sample collection  
Review QAPP

Bob Day  
P.O. Box 30273  
Lansing, MI 48909-7973  
517-335-3314

Provide SOP for processing caged fish samples  
Provide Analytical SOP for caged fish samples  
Review QAPP

Laboratories

Laboratory analyses for this project will be performed by several different laboratories. Lakeshore Engineering Services, Inc. will coordinate analytical services from a variety of separate laboratories under a contract agreement with the USACE. Lakeshore Engineering Services, Inc. will be responsible for sub-contracting for sample analysis. Both the contract laboratory and the analyzing laboratories will have sample analysis and review responsibilities on this project. Each laboratory will have their own provisions for conducting an internal QA/QC review of the data

before it is released to the U.S. Army Corps of Engineers. The laboratory contract supervisors listed below will contact the USACE project coordinator with any data concerns.

Several different laboratories will be utilized to perform all of the testing for this project. The following is a summary of the analytical work to be performed by each laboratory.

**Analyses**

Whole Sediment Toxicity Tests  
 Benthic Community Assessment  
 Bioaccumulation Testing  
 Caged Fish Tissue Analysis  
 Geotechnical Analysis  
 All Other Chemical and Physical Analyses

**Laboratory**

AScI  
 AScI  
 AScI/Lancaster Laboratories  
 Lancaster Laboratories  
 Coleman Engineering Company  
 Trace Analytical Environmental  
 Laboratories/Lancaster Laboratories

Written QA/QC reports will be filed by the analytical laboratories each time data is submitted to the USACE. Corrective actions will be reported to the USACE project coordinator along with the QA/QC report (see Sections 9). Any of the laboratories may be contacted directly by USEPA or USACE personnel to discuss QA concerns. Lakeshore Engineering Services will act as laboratory coordinator on this project and all correspondence from the laboratories should be coordinated through Lakeshore Engineering Services. Trace Analytical Laboratories and Lancaster Laboratory will perform all chemical analyses and AScI Corporation will perform the whole sediment toxicity testing. Coleman Engineering Co. will perform geotechnical testing (grain size distribution). Responsibilities of each lab and the laboratory coordinator are provided below:

***Person:***

Patricia Novak  
 Lakeshore Engineering Services  
 313-535-7882  
 pattin@lakeshoreeng.com

Aziz Khandker  
 Lakeshore Engineering Services  
 313-535-7882  
 azizk@lakeshoreeng.com

Ann Preston  
 Trace Analytical Laboratories  
 231-773-5998

***Responsibilities:***

Review final analytical report  
 Ensure Sub-Contract Laboratory Resources are available on an as-required basis  
 Review final analytical reports  
 Review Quality Assurance Plan

Perform independent technical review of final analytical reports

Coordinate/Perform chemical and physical analyses  
 Ensure Laboratory resources are available on an as-required basis  
 Review final analytical reports analyses  
 Supply required sample bottles, required preservatives, and coolers (including temperature blanks)

Al Mozol  
ASCI Corporation  
218-722-4040

Coordinate/Perform Whole Sediment Toxicity Tests  
Ensure Laboratory resources are available on an as-required basis  
Review final analytical reports analyses  
Supply Required sample bottles, required preservatives, and coolers (including temperature blanks)  
Perform *Lumbriculus* exposure to test sediments  
Ship *Lumbriculus* tissue samples to Lancaster Laboratory for analysis

Kathleen Loewen  
Lancaster Laboratory  
717-656-2300

Coordinate/Perform chemical analyses  
Ensure Laboratory resources are available on an as-required basis  
Review final analytical reports analyses  
Supply required sample bottles, required preservatives, and coolers (including temperature blanks)  
Submit a QA/QC Case Narrative

Jim Strigel  
Coleman Engineering  
906-774-3440

Coordinate/Perform geotechnical analysis  
Ensure Laboratory resources are available on an as-required basis

## 2. Project Description

### 2.1 Data Uses and Expected Measurements

GLNPO proposes a full-scale post-remediation assessment at the Ford Monroe site to augment the data collected during the 1998 assessment. The full-scale post-remediation assessment would incorporate the collection of sediment cores and surficial grabs at and downstream of the remedial site, and caged fish testing at and downstream of the remedial site. Work would be coordinated with the Michigan DEQ to insure comparability with the existing pre-remediation data. The proposed work components are summarized below.

#### Determination of Existing Pre-Remedial Data Availability

The first step of this project will be to identify sources and availability of pre-remediation data for this site. The USEPA Superfund, the Michigan DEQ, and the U.S. Army Corps of Engineers (USACE) all have collected pre-remedial data at or near this site. Both sediment data and caged fish data exist for documenting pre-remedial conditions at this site.

GLNPO will coordinate with each of these agencies to determine the quality and availability of existing data.

#### Sediment Chemistry Sampling

Sediment chemistry sampling will consist of the collection of a sediment core or a surficial sediment grab sample at approximately 20 locations. Three (3) of the locations will be upstream of the remediated area, twelve (12) of the locations will be in the remediated area, and five (5) of the locations will be downstream of the remediated area. These locations were selected to meet the priority goal of determining the success of the remediation effort, and the secondary goal of providing data on the overall state of sediment chemistry within the entire AOC.

The 12 locations within the remediated area were selected utilizing a systematic-aligned sampling algorithm in order to minimize the probability of missing a major sediment deposit (greater than 25 feet in diameter) within the remediated area.. Sampling locations outside the remediated area were selected by identifying sediment deposits within the AOC, assigning the deposits to an "upstream" or "downstream" category, and then randomly selecting five locations from "downstream" deposits, and three locations from "upstream" deposits. More locations were selected downstream of the of the remediated area, since historical sampling indicates that the Ford-Monroe Outfall was the major source of contamination within the AOC.

If insufficient sediment is present for the collection of a grab sample and/or core within the remediated area, no sample will be collected at the site, and the site will be identified as "No Sediment Present". If insufficient sediment is present for the collection of samples outside of the remediated area, the sampling crew will probe the entire sediment deposit until an adequate sampling location is identified.

All sediment cores will be sectioned into sub-samples of 0"-6", 6"-18", 18"-54", and 54"-90". GLNPO anticipates collection of an average of two (2) samples per site. All sediment samples collected will be analyzed for PCBs, using the PCB Arochlor (to be consistent with the historical data sets) and PCB congener-specific analysis as provided in USEPA SW-846 Method 8082; heavy metals (USEPA Methods 6020 and 6010B); and mercury (USEPA Method 7471A).

#### Whole Sediment Toxicity Testing

Surficial grab samples will be collected from 8 locations using a ponar dredge sampler. The sampling locations will be positioned as follows: two (2) locations upstream of the remediated area, four (4) locations within the remediated area, and two (2) locations downstream of the remediated area. Rationale and sample location algorithms for the location of toxicity sampling is similar to that described above under "Sediment Chemistry Sampling". Fewer sites were selected based on budget constraints.

These surficial grab samples will be used to conduct *Hyalella azteca* 28-day and *Chironomus tentans* 10-day whole sediment toxicity tests according to USEPA Test Methods 100.4 and 100.2, respectively, as detailed in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants in Freshwater Invertebrates* (USEPA, 2000). Sediment samples will also be analyzed for PCB concentrations, simultaneously extracted metals-acid volatile sulfide (SEM-AVS), total metals, grain size and Total Organic Carbon (TOC).

#### Benthic Community Structure Evaluations

Surficial grab samples will be collected from 8 locations using a ponar dredge sampler. The sampling locations will be positioned as follows: two (2) locations upstream of the remediated area, four (4) locations within the remediated area, and two (2) locations downstream of the remediated area. Testing manuals recommend co-locating benthic community evaluations with whole sediment toxicity testing locations to allow for examination of correlations between the data.

These surficial grab samples will be used to conduct benthic macroinvertebrate community assessments. Organisms contained in the sediment samples will be sorted and enumerated in to the following orders or families: *Oligochaeta*, *Chironomidae*, *Bivalvia*, *Gastropoda*, *Ephemeroptera*, *Odonata*, *Plecoptera*, *Hemiptera*, *Megaloptera*, *Trichoptera* *Coleoptera*,

*Diptera* (other than *Chironomidae*), *Hirudinea*, and *Amphipoda* using published taxonomic keys (e.g. Wiederholm [1983]; Merritt and Cummins [1984], Pennak [1989], Thorp and Covich [1991]). Samples will be used to estimate macroinvertebrate numerical abundance (individuals per square meter), species composition, and taxa richness. Identification will be to the lowest taxonomic level below family.

#### Sediment Bioaccumulation Tests

Surficial grab samples will be collected from 8 locations using a ponar dredge sampler. The sampling locations will be positioned as follows: two (2) locations upstream of the remediated area, four (4) locations within the remediated area, and two (2) locations downstream of the remediated area. Testing manuals recommend co-locating bioaccumulation evaluations with whole sediment toxicity testing locations to allow for examination of correlations between the data.

These surficial grab samples will be used to conduct *Lumbriculus variegatus* 28-day bioaccumulation testing for PCB congeners. Tests will be conducted according to USEPA Test Method 100.3 as detailed in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants in Freshwater Invertebrates* (USEPA, 2000).

#### Caged Fish Testing

Caged young or one year catfish (4"-8" long) testing will take place at approximately four (4) locations, plus one replicate at one of the four locations within the AOC as close as possible to MDEQ's historical caged fish sampling locations. Duration of the caged fish testing will be 28-days. At the initial startup of the sampling, four (4) day-0 fish will be sampled for the control group. After the end of the 28 days, four (4) fish from each sampling location plus another four (4) fish from the replicate cage will be sampled to make a total of twenty (24) samples collected for the caged fish testing. MDEQ will supply the cages for the study. The caged fish will be analyzed for total PCB levels using the method outlined in *Bulletin of Environmental Contamination and Toxicology*, 1986, Vol. 37, pages 1-9 (copy in Appendix I)

Prior to setting each cage, one sediment and one water column sample will be collected at each site, including the replicate location. Prior to pulling each trap, a water sample will be collected at each site. The sediment samples will be collected using a ponar grab sampler and water samples will be collected using a Van Dorn sampler. All water and sediment samples collected will be analyzed for PCB concentrations using the PCB Arochlor and congener-specific analysis (EPA Method 8082).

The Michigan DEQ has the most experience conducting caged fish testing at this site. The Michigan DEQ will assist in coordinating the caged minnow testing and corresponding sediment and water sampling at the site. The USACE contract laboratory will perform the laboratory analysis and costs will be covered using the existing Inter-Agency Agreement.

**Care should be taken to insure that caged fish testing is performed at approximately the same time of year as historical caged fish testing, which is between August and September. This testing should also be scheduled to avoid any significant dredging projects within the Raisin River watershed, though none are currently scheduled for 2002.**

### Bathymetric Survey

A sediment probe will be used to estimate the depth of sediments present at the 20 sediment coring locations. Sediment depth will be estimated to the nearest 0.25 feet.

## 2.2 Criteria and Objectives

### 2.2.1 Sediment Chemistry

Sediment Chemistry data will be compared to existing sediment quality guidelines (SQGs) like those of *MacDonald et al.* (2000) and *Persuad et al.* (1993). Table 1 provides the required reporting limits necessary to allow for sediment chemistry results to be compared directly to these screening guidelines.

**Table 1.** Screening Values for Contaminants of Concern

Analyte	Unit	Required Reporting Limits
Arsenic	mg/kg DW*	6.0
Cadmium	mg/kg DW*	0.6
Chromium	mg/kg DW*	26.0
Copper	mg/kg DW*	16.0
Lead	mg/kg DW*	31.0
Mercury	mg/kg DW*	0.2
Nickel	mg/kg DW*	16.0
Zinc	mg/kg DW*	120.0
Total PCBs (as.Arochlors)	mg/kg DW*	0.676
PCB Congeners	mg/kg DW*	0.01

\* DW = Dry Weight

### 2.2.2 Fish Tissue Chemistry and *L. variegatus* Tissue Chemistry

Fish tissue and *L. variegatus* tissue chemistry analysis must be of sufficient quality to allow for comparison to historical and future data collected at the site. Table 2 provides the required reporting limits necessary to allow for fish tissue chemistry results to be compared directly to historical and future data.

**Table 2.** Required Reporting Limits for Tissue Chemistry to Allow Comparison to Historical and Future Data Sets

<b>Analyte</b>	<b>Units</b>	<b>Required Reporting Limits</b>
Total PCBs (as Arochlors)	mg/kg	0.30
PCB congeners		
<i>Level of Chlorination</i>		
Mono- to tri-chloro	mg/kg	0.02
Tetra- to Hexa-chloro	mg/kg	0.03
Hepta- to Octa-chloro	mg/kg	0.04
Nona- to Deca-chloro	mg/kg	0.05

### 2.3 Special Personnel, Training, and Equipment Requirements

#### Sediment Sampling

Sediment sampling will require the use of the USEPA's Research Vessel (R/V) Mudpuppy or an equivalent vessel. Additional equipment requirements for collecting sediment core and sediment ponar samples are contained in Appendix A.

#### Benthic Community Assessment

Collection and filtering of sediment samples for benthic community assessments requires the use of an elutriator fitted with a 500 micron mesh filter (equipment available on the R/V Mudpuppy). Additionally, a 0.500 M formaldehyde solution is required for preserving the benthic community samples. A detailed SOP is provided in Appendix B.

#### Caged Fish Sampling

Collection of caged fish samples will follow the SOP provided in Appendix C in order to be consistent with historical sampling performed by the MDEQ. Training and equipment requirements are provided in Appendix C.

### 2.4 Project Schedule

A tentative project schedule is provided in Table 3. All personnel shown in Figure 1 should be contacted regarding significant schedule changes.

**Table 3. Tentative Project Schedule**

<b><u>Task</u></b>	<b><u>Completion Date</u></b>
QAPP Development and Sign-Off	August 31, 2001
Sediment Sampling (chemistry, toxicity, bioaccumulation, and benthic community)	October 2001
Completion of Sediment Analysis and Testing	January 2002
Analytical Report Due to USACE	March 2002
Sediment Sampling Summary Report and Analytical Data Due to USEPA-GLNPO	April 2002
Caged Fish Sampling	August/September 2002
Sediment and Water Sampling at Caged Fish Sites	August/September 2002
Completion of Caged Fish, Sediment, and Water Analytical Analysis	October/November 2002
Caged Fish Sampling Summary and Analytical Data Report Due to USEPA GLNPO	December 2002

### **3. Sampling Plan**

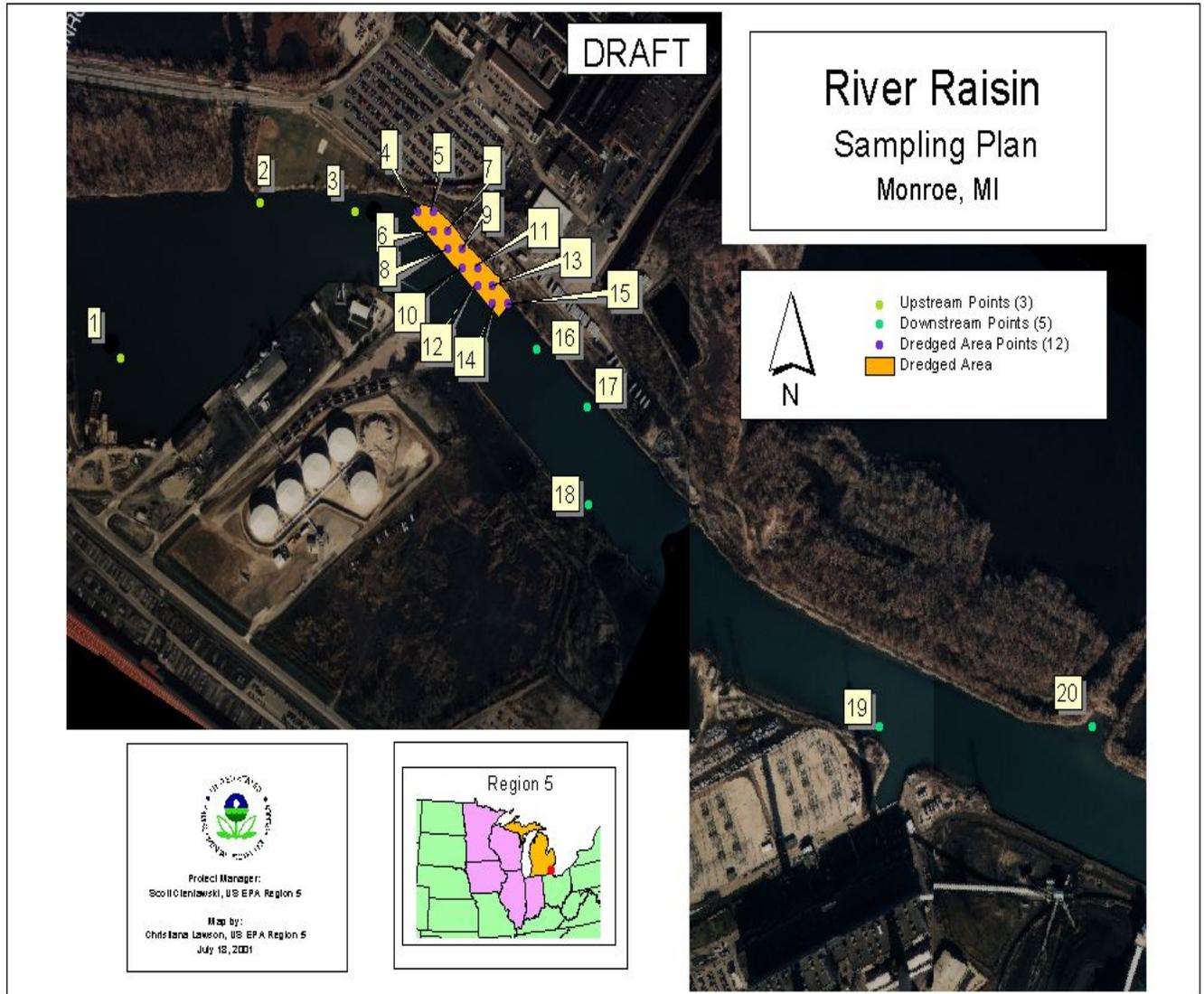
#### **3.1 Sampling Network Design and Rationale**

The purpose of this sampling survey is to determine the quality of the sediments and health of the aquatic ecosystem in the vicinity of a previously completed sediment remediation project as well as to provide an overall summary of sediment quality within the Raisin River AOC. In order to obtain a full picture of the health of the aquatic ecosystem data on a large number of ecosystem metrics need to be collected. Sediment chemistry, sediment toxicity, in-situ bioaccumulation, and ex-situ bioaccumulation samples will be collected along with water and benthic community samples. These samples will allow us to determine the levels of contaminants present in the water, the direct impact of sediments on the benthic community, and the potential for bioaccumulation of contaminants through the aquatic food chain.

Figure 2 presents an overview of the assessment area, a delineation of the remediated area, and approximate locations for collection of sediment samples. Approximate latitude/longitude of the sampling points and specific analytical tests to be performed on each sample are provided in Appendix K

The sampling locations are designed to provide focused coverage of the removal area as well as some general coverage of areas upstream and downstream of the removal area. In-depth rationale and algorithms used for selecting sampling locations are provided in Section 2.1. Table 5 summarizes the types of data and analyses to be collected at each type of sampling location.

**Figure 2. Sampling Locations**



**Table 4. Summary of Data and Analyses at Sampling Locations**

<b><i>Core Samples (20 Locations)</i></b>	<b><i>Ponar Samples (8 Locations)</i></b>
Sediment Chemistry	Sediment Chemistry
Sediment Depth	Whole Sediment Toxicity
Water Depth (corrected to LWD)	Benthic Community
Latitude/Longitude	Water Depth (corrected to LWD)
	Latitude/Longitude
<b><i>Caged Fish Sample (4 Locations)</i></b>	<b><i>Bioaccumulation Samples (8 Locations)</i></b>
Sediment Chemistry	Tissue Chemistry
Water Chemistry	Latitude/Longitude
Fish Tissue Chemistry	
Latitude/Longitude	

### 3.2 Definition of Sample Types

Four types of sediment samples will be collected during this survey; Routine Field Samples (RFS), Field Replicates (FR), Field Duplicates (FD), and Matrix Spikes/Matrix Spike Duplicates (MS/MSD). Each sample type is described below.

***Routine Field Samples (RFS):*** Prepared by collecting a single ponar grab sample or section of a sediment core, homogenizing the sediments collected, and filling all required sample jars. Routine field samples will be collected at twenty (20) locations. Locations of the RFS are indicated in Appendix K.

***Field Duplicates (FD):*** Prepared by filling a second set of sample jars from a single ponar or section of a sediment. FDs will be collected at three (3) sediment core locations and one (1) ponar sampling location. This is approximately equivalent to a ratio of FDs to RFSs of 1 to 10 (10%). Locations of the FDs are indicated in Appendix K.

***Field Replicates (FR):*** Prepared by collecting a second, separate ponar grab or sediment core sample, homogenizing the material separately from the RFS and filling the required sample bottles jars. FRs will be collected at two (2) sediment core locations, one (1) ponar sampling, and one (1) caged fish location. This is approximately equivalent to a ratio of FRs to RFSs of 1 to 10 (10%). Locations of the FRs are indicated in Appendix K.

***Matrix Spike/Matrix Spike Duplicates (MS/MSD):*** The MS/MSD samples will be collected out of the same sample as the RFS, homogenizing the sediment sample, and filling the required sample jars. One MS/MSD sample will be collected from each of six (6) sediment ponar locations. This is approximately equivalent to a ratio of 1 to 10 (10%) since a total of at least sixty samples will be collected for this project. Locations of MS/MSDs are indicated in Appendix K.

The tables in Appendix K summarize the types of samples to be collected and analyzed for each sampling location.

### 3.3 Type and Number of Samples

Table 5 summarizes the type and number of samples to be collected during this sampling event. The estimated number of samples include all RFS, FD, FR, and MS/MSD samples.

**Table 5. Summary of Type and Number of Samples to be Collected**

Sample Type	Estimated Number of Samples	Sample Matrix	Analysis Required
Sediment Chemistry from Cores	48	Sediment	PCBs (Aroclors and Congeners), Oil & Grease, TOC, % Moisture
Sediment Chemistry from Ponars	11	Sediment	PCBs (Aroclors and Congeners), SVOCs (PAHs), Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Se, Ag, and Zn), TOC, SEM-AVS, Grain Size, Nutrients (Total Phosphorus, Ammonia-Nitrogen, TKN), Moisture Content
Sediment Toxicity	11	Sediment	28-Day <i>H. azteca</i> Survival and Growth (weight and length), 10-day <i>C. tentans</i> Survival and Growth (weight)
Benthic Community	11	Sediment	Identified to Family Level
Caged Fish Tissue	24	Tissue	PCBs (Aroclors and Congeners)
Sediment from Cage Fish Sites	6	Sediment	PCBs (Aroclors and Congeners), Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Se, Ag, and Zn), TOC, Moisture Content
Water from Caged Fish Sites	12	Water	PCBs (Aroclors and Congeners), Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Se, Ag, and Zn), TOC
Sediment Bioaccumulation	11	Sediment	PCBs (Congeners)

All of the data listed in Table 5 is considered critical to the success of this assessment project.

### 3.4 Field Data Collection

SOPs for all field measurements are contained in Appendix D. Two pieces of field data will be collected that are critical to the data quality objectives for this project.

*Latitude/Longitude Location:* This data is critical for use in determining where sediment samples were collected. The two (2) Differential Global Positioning Systems (DGPS) onboard the R/V Mudpuppy are both capable of ascertaining horizontal locations with < 5 meters of accuracy. To achieve this accuracy, it is important that the DGPSs are in good working order and are obtaining strong satellite signals. The field team will be responsible for checking the satellite signal strength for the DGPS systems prior to recording this data and for ensuring that the two systems are recording equivalent horizontal locations. Any problems with signal strength or differences between the two systems shall be recorded on in the field sample log (Appendix E). If problems are noted, the field team should provide a qualitative description of the sampling location utilizing any available, permanent landmarks. Both DGPS units will have their accuracy checked prior to each days sampling activities by locating one of the USACE survey markers shown on Appendix F. The DGPS unit's antennas will be located as close to the marker as possible and the reading will be compared to those in Appendix F.

*Sediment Depth:* Sediment depth data is critical for determining the volume of sediments and mass of PCBs remaining in the removal area.

Other field data to be collected includes:

1. Water Depth (corrected to Low Water Datum post-sampling),
2. Log of Major Ship Traffic in the Survey Area,
3. Log of Significant Navigation Dredging in the Survey Area, and
4. Sediment Physical Observations.

However, these additional pieces of data are not considered critical to the objectives of this project.

### *Water Depths*

Water depths will be taken directly over the location of the sampling site prior to sample collection with a weighted measuring tape. Water depths will be reported as actual depth measured and as water depth corrected to Low Water Datum. Low Water Datum is available for Raisin River at the closest daily and hourly water levels station for Lake Erie at Fermi Power Plant, which can be obtained from the Internet at the NOAA home page for water elevations.

[Note: Low Water Datum is available in 6 minutes intervals. The address is: [http://www.opsd.nos.noaa.gov/data\\_res.html](http://www.opsd.nos.noaa.gov/data_res.html). From this address under Preliminary Water Level Data select Great Lakes Stations then choose Fermi power Plant and display recorded water levels in feet.]

## **4. Sample Collection and Handling**

### **4.1 Sample Collection**

#### **4.1.1 Sediment Cores**

Sediment cores will be collected utilizing the vibracorer sampling device located on the USEPA-GLNPO's sampling vessel, the *R/V Mudpuppy*. The vibracorer is capable of collecting continuous sediment cores up to 15 feet in length. Appendix A contains the equipment needs, the Standard Operating Procedures (SOP), and the decontamination procedures for the collection of sediment core samples during this sediment survey.

All sediment cores will be analyzed for sediment chemistry as summarized in Table 5 and explained in detail in Section 5.

#### **4.1.2 Sediment Ponars**

Sediment ponar samples will be collected utilizing the ponar dredge sampling device on the USEPA-GLNPO's sampling vessel, the *R/V Mudpuppy*. The ponar dredge sampler collects a surficial sediment sample of approximately six inches (6") in depth. Appendix A contains the equipment needs, the Standard Operating Procedures (SOP), and the decontamination procedures for the collection of sediment ponar samples during this sediment survey.

Sediment ponar samples will be used for sediment chemistry analysis, whole sediment toxicity testing, laboratory bioaccumulation testing, and benthic community analysis as summarized in Table 5 and explained in detail in Section 5.

#### 4.1.3 Caged Fish Samples

Caged fish samples will be collected according to the Standard Operating Procedures contained in Appendix C. Appendix C also contains the SOP for collecting the surficial sediment and water samples that need to be collected in conjunction with the caged fish sampling. Locations of the caged fish testing will be selected by the MDEQ to correspond to their historical sampling locations, but may be modified slightly to account for current site conditions. The MDEQ will determine the latitude/longitude locations of the caged fish samples after the cages have been set. Latitude and longitude will be recorded in a field notebook, along with the site number (i.e. C-1, C-2, etc.) and relayed to Scott Cieniawski via e-mail.

Fish tissue samples, sediment from caged fish sites, and water from caged fish sites will be analyzed for chemistry as summarized in Table 5 and explained in detail in Section 5.

## 4.2 Sample Handling

### 4.2.1 Sample Containers

After processing, sediment samples will be placed into the appropriate sample containers as summarized in Table 6. A field sample log shall be filled out for each sampling location.

*Note: The analyzing laboratory will supply all required sample containers, preservatives, and sample coolers, including a temperature blank with each sample cooler. **The coolers, sample bottles, and required preservatives for all samples, except the fish tissue samples, shall be shipped to the following address no later than September 30, 2001:***

Scott Cieniawski  
USEPA-GLNPO  
77 W. Jackson Blvd. (G-17J)  
Chicago, IL 60604

*Logistics for the delivery of the fish tissue sample containers will be made between the MDEQ, USEPA, USACE, and the analyzing laboratory prior to commencement of the caged fish sampling.*

**Table 6. Sample Container and Preservation Requirements**

<b>Analyses</b>	<b>Container</b>	<b>Preservation Technique</b>	<b>Holding Times</b>
PCBs	8 oz. Widemouth Glass	Cool/dark, $\leq 4^{\circ}\text{C}$	14 days/40 days**
SVOCs (PAHs)	4 oz. Widemouth Glass	Cool/dark, $\leq 4^{\circ}\text{C}$	14 days/40 days**
Mercury	Included in metals	Cool/dark, $\leq 4^{\circ}\text{C}$	28 days*
Metals (Cd, Cr, Cu, Ni, Pb, Se, Zn)	4 oz, Widemouth Glass	Cool/dark, $\leq 4^{\circ}\text{C}$	6 months*
AVS/SEM	4 oz., Widemouth Glass with Teflon liner	Cool/dark, $\leq 4^{\circ}\text{C}$ , No head space	7 days*
Nutrients, TOC	Included in metals	Cool/dark, $\leq 4^{\circ}\text{C}$	28 days*
Total Solids	Included in metals	Cool/dark, $\leq 4^{\circ}\text{C}$	7 days*
Percent Moisture	Included in metals	Cool/dark, $\leq 4^{\circ}\text{C}$	40 days*
Particle Size	1 Quart Zip Lock Baggies	Sealed container Do Not Cool	6 months*
Whole Sediment Toxicity	4 L, Plastic	Cool/dark, $\leq 4^{\circ}\text{C}$	14 days
L. variageus, Bioaccumulation	4 L, Plastic	Cool/dark, $\leq 4^{\circ}\text{C}$	14 days
Benthic Community Assessment	1 L Plastic	Cool/dark, $\leq 4^{\circ}\text{C}$ , airtight, preservative of formalin	14 days
Fish Tissue	aluminum packages	Frozen/dark, $\leq 4^{\circ}\text{C}$	14 days/40 days**

\* From time of collection to analysis

\*\* From time of collection to extraction/From time of extraction to analysis

#### 4.2.2 Sample Labeling

Each sample bottle shall be individually labeled using a waterproof pen. The label shall contain the following information:

- **Unique Sample Number:** RR01-XX-A; where “RR01” refers to the Raisin River 2001 sampling event, “XX” refers to the numerical sequence of the sample locations, and “A” refers to the alphabetical sequence indicating sample depth (“A” is 1<sup>st</sup> layer/segment, “B” is the 2<sup>nd</sup> layer/segment, etc., a ponar sample will be designated “P”) Field duplicates and field replicates shall receive their own unique sample number so as to provide a blind duplicate/replicate for laboratory analysis. Caged fish sampling locations are numbered C-1, C-2, etc. Therefore caged fish samples will be labeled RR01-C-1, RR01-C-2, etc.)
- **Sample Date** (MM-DD-YYYY)
- **Sample Time** (HH:MM, on a 24-hour clock)
- **Analysis to be performed** (e.g. PCBs, metals, whole sediment toxicity, etc.)
- **Sampler's Initials**

An example label is shown in Figure 3. Clear tape will be placed over the label after the label has been completely filled out and attached to the sample container. The sample identification number and date of sample collection will be written on the sample container closure with a water proof marker.

**Figure 3. Example Sample Label**

<b>RR01-09-A</b>	10-10-2001
	13:30
<b>PCBs</b>	SEC

#### 4.2.3 Shipment and Chain-of-Custody

After collection and labeling, all glass containers shall be placed in a zip-lock bag, placed in an appropriate sample cooler. Within 24 hours of sample collection, the samples will be sent to the respective analyzing laboratory. After samples are collected each day, the USEPA Field Team Leader shall be responsible for shipping and/or arranging pickup of samples. The Field Team Leader shall insure that:

1. The coolers contain sufficient ice to keep the sample below 4° C during the shipment process,
2. Are immobilized with bubble pack to reduce the risk of breakage,
3. The chain of custody form (see example in Appendix G) is properly filled out,
4. A copy of the chain-of-custody form shall be retained and provided to the project manager,
5. A copy of the chain-of-custody form will be placed in a "ziploc" bag and taped to the inside lid of the cooler,
6. A copy of the chain-of-custody form is faxed to the Lakeshore Engineering Services Project Manager at (313) 535-7875,
7. A temperature blank is included in each sample cooler (temperature blank to be supplied by the laboratories),
8. The outside of the container will be shut using fiberglass or duct tape,
9. The laboratory name and address, as well as the return name and address, will be clearly labeled on the outside of the container,
10. These samples will be sent to the contract laboratory by an overnight courier, and
11. Receipts of bills of lading will be retained as part of the permanent documentation.
12. Commercial couriers are not required to sign off on the sample tracking form as long as it is sealed inside the sample cooler.
13. Laboratories are contacted prior to shipment to insure they are prepared for sample arrival.
14. Whole fish samples and *L. variageus* (bioaccumulation) samples are shipped frozen.

**Note:** *Each analyzing laboratory will supply chain-of-custody forms to the USEPA field team leader prior to the sampling event.*

Table 7 summarizes where each of the respective types of samples shall be shipped.

**Table 7. Addresses for Shipment of Samples**

<u>Analysis Type</u>	<u>Laboratory Contact Information</u>
PCB aroclors and congeners Sediment, water and tissue SEM & AVS	Kathleen Loewen Lancaster Laboratory 2425 New Holland Pike Lancaster, PA 17605-2425 phone: (717) 656-2300
Metals (including Hg), Nutrients, TOC, PAHs, % Moisture	Ann Preston Trace Analytical Laboratories 2241 Black Creek Road Muskegon, MI 49444-2673 phone: (231) 773-5998 ext. 224
Whole Sediment Toxicity, Benthic Community Assessment, and Bioaccumulation Exposure	Al Mozol ASCI Corporation 4444 Airpark Blvd Duluth, MN 55811 phone: (218) 722-4040
Caged Fish Samples (PCB Aroclors and Congener Analysis)	Lancaster Laboratory/Sample Administration Grp. 2425 New Holland Pike Lancaster, PA 17605-2425 phone: (717) 656-2300
Grain Size	Coleman Engineering - Jim Strigel 635 Industrial Park Road Iron Mountain, MI 49801 Phone: (906) 774-3440

#### 4.2.4 Receipt of Samples

Upon receipt of project samples, each laboratory shall

- Complete their portion of the chain-of-custody forms,
- Contact the Lakeshore Engineering Services Project Manager to inform her of sample receipt and to discuss any problems or issues,
- Insure that the samples are maintained at < 4°C,
- Complete a Cooler Receipt Form (See example in Appendix H).
- If there are any sample shipment problems, the laboratory should contact Lakeshore Engineering Services Project Manager (Patricia Novak) and the Lakeshore Engineering Services Project Manager shall contact USEPA Project Manager (Scott Cieniawski) as soon as the sample shipment problem is discovered,
- Fax a copy of the chain-of-custody form to the USEPA project manager, Scott Cieniawski, at 312-353-2018.

## 5. Laboratory Analysis

### 5.1 Analysis Methods

Analysis and preparation methods for all required analyses are provided in Table 8.

**Table 8. Laboratory Analysis and Preparation Methods**

<i>Analyte</i>	<i>Analysis Method</i>	<i>Sample Preparation Method</i>	<i>Sample Cleanup Method</i>	<i>Laboratory SOP</i>
Moisture Content (Total Solids)	ASTM D2937	N/A		Per ASTM
Grain Size without hydrometer	ASTM-D422	N/A		Per ASTM
28-day <i>H. azteca</i> , and 10-day <i>C. tentans</i> Whole Sediment Toxicity Tests	EPA/600/R-99/064, Methods 100.4 (Survival & Length) and 100.2 (Survival & Weight)	N/A		S-301 and S-304
TOC	9060/Lloyd-Kahn	N/A		UWC-SOP-KAHN
Ammonia-Nitrogen	E350.1	N/A		UWC-SOP-350.2
TKN	E 351.2	N/A		UWC-SOP-351.3
Total Phosphorus	E 365.2M	N/A		UWC-SOP-365.2
Metals Kit	EPA 6020, 6010A (As & Cd analyzed using 6010B ICAP Trace procedures), 7471A	EPA 3051 for sediments and EPA 3015 for water		UME-SOP-6010B-T, UME-SOP-245.1
PCBs (Sediments) Aroclors & Congeners <sup>(1)</sup>	EPA 8082B	EPA 3540C	EPA Method 3620B, 3665A, or 3630C	UGE-SOP-8082
PCBs (Fish Tissue)	EPA 8082B	Lancaster Analysis #2487 "Food and Tissue Preparation"		UGE-SOP-8082
Oil and Grease	EPA 9070 for water EPA 9071A for sediments	N/A		
PCBs (Water)	EPA 8082B	EPA 3510C	EPA 3630C	
PAHs	EPA 8270C	EPA 3540C	EPA Method 3630C	
Acid Volatile Sulfide (AVS) <sup>(2)</sup>	EPA-121-R91-100	Acid Leach		EPA-121-R91-100
Simultaneously Extracted (SEM) Metals <sup>(2)</sup>	EPA 6020M 6010A	N/A		EPA-121-R91-100

<sup>(1)</sup>The following 19 PCB congeners, listed by their International Union of Pure and Applied Chemistry Number (IUPAC #), will be analyzed and reported for sediment chemistry: 1, 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, 206.

<sup>(2)</sup> AVS-SEM Analysis Method can be found in the "Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment", EPA#: 821/R-91-100 YEAR: 1991, NTIS#:PB93-155901, ERIC#: D-121

## 5.2 Data Quality Objectives (DQOs)

Data from the historical sampling events contains very little information regarding data quality objectives. Additionally, the analytical detection limits obtained in the historical sampling events (i.e., PCB Aroclor detection limits) may not be sufficient to meet the secondary objectives of this project. Therefore, the DQOs chosen for this project will be based on the objectives required to adequately assess the current state of the aquatic system in the study area.

The DQOs for the laboratory analysis portion of this project are defined according to the following six quality assurance objectives.

### Definitions

*Instrument Detection Limit (IDL):* The instrument detection limit (IDL) is the lowest analyte concentration that an instrument can detect. The IDL is determined on samples that have not gone through any sample preparation (e.g. calibration standards).

*Limits of Quantification (LOQ):* The limits of quantification is the lowest analyte concentration that can be accurately measured and reported, as opposed to simply detected.

*Method Detection Limit (MDL):* Method detection limits (MDL) will be determined by making repeated measurements (a minimum of seven) over several non-consecutive days of either a calibration blank or a low-level standard with a concentration within 1-5 times the IDL. The MDL is calculated, at the 95 percent confidence level, as 3 times the standard deviation of the measured sample concentrations.

*Target Detection Limit (TDL):* The target detection limit (TDL) is the concentration at which each analyte must be detected and quantified in order to meet the study objectives. This means that, if possible, all IDLs, MDLs and LOQs, should be less than the TDLs for all analytes. If the laboratory expects any of the IDLs, MDLs, or LOQs to exceed the required TDLs, they must contact the USACE and USEPA project managers to develop corrective action procedures.

### 5.2.1 Method Detection Limits and Level of Quantification

For quantitative physical and chemical analyses, analytical laboratories will be required to determine the instrument detection limit (IDL) prior to any analysis of the routine samples. The target detection limit (TDL) is the concentration at which the presence of an analyte must be detected to properly be able to assess and satisfy the DQOs. To be acceptable, a laboratory must demonstrate that the MDL is less than or equal to the TDL through use of laboratory quantitation standards. The laboratories shall also strive to set the dry sample Limits of Quantification (LOQs) below the applicable TDLs. Tables 1 and 2 contain the threshold effect concentrations (TECs) for the chemicals to be analyzed that have actually had the TECs calculated. Tables 9 and 10 contain this exact information, plus a few additional parameters that do not have calculated TECs, which are all also listed at the TDL for each parameter.

Target detection limits for all required sediment chemistry and tissue chemistry are provided in Tables 9 and 10, respectively.

**Table 9. Target Detection Limits for Sediment Chemistry**

Analyte	TDL	Unit
Arsenic <sup>(1)</sup>	1.2	mg/kg DW
Cadmium <sup>(1)</sup>	0.2	mg/kg DW
Chromium	5.0	mg/kg DW
Copper	3.0	mg/kg DW
Lead	6.0	mg/kg DW
Mercury	0.05	mg/kg DW
Nickel	3.2	mg/kg DW
Zinc	20.0	mg/kg DW
Total Organic Carbon	1000.0	mg/kg DW
PCBs, as Aroclors <sup>(2)</sup>	0.1	mg/kg DW
PCBs, as Congeners <sup>(2)</sup>	0.01	mg/kg DW
PAHs	0.6	mg/kg DW
Acid Volatile Sulfides	50.0	mg/kg DW
TKN	20.0	mg/kg DW
Total Phosphorus	5.0	mg/kg DW
Ammonia Nitrogen	1.0	mg/kg DW

<sup>(1)</sup> Analysis may require EPA Method 6010B ICAP trace procedures to achieve required stated detection limits.

<sup>(2)</sup> Analysis may require additional sample cleanup preparation to achieve required detection limit.

<sup>(3)</sup> Metals not listed above to complete the 13 Metals Kit (Ba, Fe, Mn, Se, and Ag) should have the assigned laboratory meet the lowest detection limit possible.

**Table 10. Target Detection Limits for Tissue Chemistry**

Analyte	Units	TDL
Total PCBs (as Aroclors)	mg/kg	0.30
PCB congeners		
<i>Level of Chlorination</i>		
Mono- to tri-chloro	mg/kg	0.02
Tetra- to Hexa-chloro	mg/kg	0.03
Hepta- to Octa-chloro	mg/kg	0.04
Nona- to Deca-chloro	mg/kg	0.05

**Note:** If a laboratory is unable to obtain MDLs and LOQs that are below the respective TDLs for each analyte, the laboratory shall contact the U.S. Army Corps of Engineers Project Coordinator and/or the U.S. Environmental Protection Agency's Project Manager to discuss required course of action. Decisions to be made could include: implementation of additional

sample clean-up procedures prior to analysis, USEPA acceptance of higher MDLs and LOQs, or implementation of other potential suggestions.

**Note:** It is understood that potential high moisture contents of the sediments could impact MDLs and LOQs achieved by the laboratory. In an effort to reduce the impact of high water content on MDLs and LOQs the labs shall decant free water from the surface of the sediment samples prior to analysis. Tentative laboratory Reporting Limits are listed in Tables 9 and 10.

### 5.2.2 Bias

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Bias assessments for environmental measurements are made using personnel, equipment, and spiking materials or reference materials as independent as possible from those used in the calibration of the measurement system. When possible, bias assessments should be based on analysis of spiked samples rather than reference materials so that the effect of the matrix on recovery is incorporated into the assessment. A documented spiking protocol and consistency in following that protocol are important to obtaining meaningful data quality estimates. Spikes should be added at concentrations approximately at the mid-range. Spiked samples shall be used in accordance with the specified method.

Bias will be assessed through the use of certified reference materials (CRMs), standard reference materials (SRMs: a reference material certified by the U.S. National Institute of Standards Technology [U.S. NIST]), or other standards, such as, matrix spikes. The use of spiked surrogate compounds for GC and GC/MS procedures for PCB and PAH compounds, respectively, will be used to assess for bias.

Matrix spike and matrix spike duplicate samples (MS/MSD) also will be used to assess bias as prescribed in the specified methods. Acceptable recovery values will be within the recoveries specified by each of the analysis methods. Control samples for assessing bias will be analyzed at a rate as specified in the analytical SOPs and specified analytical methods.

### 5.2.3 Precision

Precision is a measure of agreement among replicate measurements of the same property, under prescribed similar conditions. This agreement is calculated as either the range (R) or as the standard deviation (s). It may also be expressed as a percentage of the mean of the measurements, such as relative percent difference (RPD) or relative standard deviation (RSD) (for three or more replicates).

Laboratory precision is assessed through the collection and measurement of laboratory duplicates. The laboratories shall follow the protocols in the specified method and corresponding SOPs regarding the frequency of laboratory duplicates. This allows intra-laboratory precision information to be obtained on sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision. An estimate of long-term precision can be obtained by separating the two samples and processing them at

different times, or by different people, and/or analyzed using different instruments. Acceptable RPDs will be in accordance to those specified by each analysis method.

For duplicate measurements, relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100\%$$

RPD = relative percent difference

D<sub>1</sub> = sample value

D<sub>2</sub> = duplicate sample value

For three or more replicates:

$$RSD = (s/x) \times 100$$

RSD = relative standard deviation

s = standard deviation of three or more results

x = mean of three or more results

Standard deviation is defined as follows:

$$s = ((\sum(y_i - \text{mean } y)^2 \times 1/(n-1)))^{0.5}$$

s = standard deviation

y<sub>i</sub> = measured value of the replicate

mean y = mean of replicate measurements

n = number of replicates

Additionally, precision will be assessed by the collection of field duplicates and field replicates. Field duplicates are collected by splitting samples AFTER the homogenization process. The duplicates are assigned a separate Sample ID number and, therefore, provide a blind measure of the laboratories precision by providing the laboratory with two basically identical samples. Field duplicates measure the ability for the laboratory to obtain similar results from two separate, blind samples. RPD for field duplicates should meet the RPD control limits specified in Table 11.

Field replicates are collected from slightly different locations (<3 feet away) than the original sample. Field replicates provide a measure of the variability inherent in the entire sampling and analysis process, including, small-scale variability of site conditions, consistency of sampling and homogenization process, and laboratory analysis. The field replicates provide a general picture of the amount of variability that can be expected between this and future sampling events, even if site conditions do not change substantially. This is an important consideration since this data will be compared to historical and future sampling events. Since site variability can greatly influence RPD for field replicates, no strict RPD measures will be used to evaluate this measure. However, most sediment guidance recommends that RPD measures for field replicates be in the same range as that for field duplicates.

Quality control limits for Precision, Accuracy, and Completeness are summarized in Table 11.

**Table 11. Quality Control Limits (Aqueous and Sediment Matrices)**

Analyte	Precision (RPD)	Accuracy (%)	Completeness (%)
Individual PAHs	≤40	<i>As determined by Laboratory</i>	90%
PCBs (Congeners and Aroclors)	≤50	<i>As determined by Laboratory</i>	90%
Mercury	≤40	<i>As determined by Laboratory</i>	90%
Metals (As, Cd, Cr, Cu, Ni, Pb, Zn)	≤35	<i>As determined by Laboratory</i>	90%
Acid Volatile Solids	≤40	<i>As determined by Laboratory</i>	90%
Simultaneously Extracted Metals (Cd, Cu, Pb, Ni, Ag, Zn)	≤40	<i>As determined by Laboratory</i>	90%
Percent Moisture	As specified in method	<i>As specified in method</i>	90%
Particle Size	As specified in method	N/A	90%
Ammonia	≤25	80-120	90%
TOC	≤20	50-130	90%
Toxicity Testing		>80% Mean Survival of Negative Control Samples	90%

RPD = Relative Percent Difference

#### 5.2.4 Accuracy

Accuracy measures how close analytical results are to a true or expected value. Accuracy objectives will be determined by calculating the percent recovery range of laboratory matrix spikes and matrix spike duplicates. Accuracy measures are calculated using the RPD between the expected value and the actual analytical results.

#### 5.2.5 Representativeness

Representativeness is the degree to which the sampling data properly characterize the study environment. For the field-sampling phase, the previously established sampling sites reasonably cover the entire AOC, and have been previously deemed to adequately represent the various sub-units within the AOC. A statistical analysis of the 12 sampling locations designed to cover the remediated area, indicates a less than 20% probability of missing a significant sediment deposit of over 25-feet in diameter. This probability is deemed acceptable for this project.

Additionally, due to the mobile, semi-fluid nature of sediment deposits over time, the sediment within a particular depositional zone tend to be stratified vertically (as a surrogate of time) rather than horizontally. However, sediments from one depositional zone can vary drastically from sediments in other depositional zones based on location and time of contamination release.

Therefore, by randomly sampling sediment deposits from a range of spatial locations within the AOC we obtain a representative description of sediment quality within the AOC.

In the analytical phase, and as specified elsewhere in this document, appropriate sample storage and preservation, and sample homogenization will insure that the samples analyzed adequately reflect conditions as they existed in the natural environment.

### **5.2.6 Comparability**

Comparability states the confidence with which one data set can be compared to another. Comparability will be enhanced by the consistent use of standardized sampling methods and specified protocols for the sampling phase and through the use of standard documented methodologies for analyte determinations. Any deviations from the standardized, selected methods or protocols will be clearly documented by the laboratories and noted in the final analytical report. There are a number of issues that can make two data sets comparable, and the presence of each of the following items enhances their comparability:

- Two data sets should contain the same set of variables of interest
- Units in which these variables were measured should be convertible to a common metric
- Similar analytical procedures and quality assurance should be used to collect data for both data sets
- Time measurements of certain characteristics (variables) should be similar for both data sets
- Measuring devices used for both data sets should have approximately similar detection levels
- Rules for excluding certain types of observations from both samples should be similar
- Samples within data sets should be selected in a similar manner
- Sampling frames from which the samples were selected should be similar
- Number of observations in both data sets should be of the same order or magnitude.

These characteristics vary in importance depending on the final use of the data. The closer two data sets are with regard to these characteristics, the more appropriate it will be to compare them. Large differences between characteristics may be of only minor importance, depending on the decision that is to be made from the data.

For this investigation, comparability will be satisfied by ensuring that the field sampling plan is followed, standard EPA Methods of analysis are used for sample analysis and that proper sampling techniques are used.

### **5.2.7 Completeness**

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that the project expected to be obtained under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness objectives for this project will be greater than 90%. Laboratory completeness is a measure of the amount of valid measurements obtained

from all the measurements taken in the project. Laboratory completeness for this project will be greater than 90% of the total number of samples submitted to the analytical laboratories.

The calculation for percent completeness is as follows:

$$\%C = 100\% \times (V/n)$$

%C = percent completeness

V = number of valid<sup>(1)</sup> measurements

n = number of measurements planned

<sup>(1)</sup> For this sampling event, a valid measurement is defined as the arrival at a sampling location and collection and analysis of a sediment sample. However, since remediation would be expected to remove all sediment that is present, for the remediated area only, a valid measure is collection and analysis of a sediment sample or the analysis of sediment depth and the recording of "No Sediment Present".

## **6. Documentation and Records**

### **6.1 Field Documentation**

Field logs, ship logs, and chain of custody documents will be used to record appropriate sample collection information in the field.

Sediment Sample Collection Logs: An example sediment sample collection log is provided in Appendix E. A sediment sample collection log will be filled out by the field crew for each sample collected. All original field data sheets shall be turned over to the Project Coordinator at the conclusion of the field sampling and shall be kept as part of the permanent project file.

Ship Log: A ship log maintaining a summary of sample collection information shall be maintained for each day of field sampling. Information to be included in the ship log shall include: sample location ID, latitude/longitude of each sampling location, time of sample collection. The ship log shall remain with the ship files for a period of at least 2 years following the conclusion of field sampling.

#### Chain-of-Custody Forms:

An example chain of custody form is provided in Appendix G. A chain-of-custody form will be filled out for each set of samples shipped to the laboratory. A copy of the chain-of-custody form will be faxed to the Lakeshore Engineering Services Project Manager at the end of each field day. All copies of the chain-of-custody form will be returned to the PI at the conclusion of the project.

### **6.2 Laboratory Reports**

All laboratory data and records will be included in the final analytical report submitted to the project manager. A complete copy of the QAPP will be provided to the labs. The project manager will be responsible for maintaining the reports in the permanent project file. The following laboratory-specific records will be compiled by the appropriate laboratory and included in the final analytical report submitted to the project manager.

Sample Data. These records contain the times that samples were analyzed to verify that they met holding times prescribed in the analytical methods. Included should be the overall number of samples, sample location information, any deviations from the SOPs, time of day, and date. Corrective action procedures to replace samples violating the protocol also should be noted.

Sample Management Records. Sample management records document sample receipt, handling and storage, and scheduling of analyses. The records verify that sample tracking and proper preservation were maintained, reflect any anomalies in the samples (such as receipt of damaged samples), note proper log-in of samples into the laboratory, and address procedures used to ensure that holding time requirements were met.

Test Methods. Unless analyses are performed exactly as prescribed by SOPs, this documentation will describe how the analyses were carried out in the laboratory. This includes sample preparation and analysis, instrument standardization, detection and reporting limits, and test-specific QC criteria. Documentation demonstrating laboratory proficiency with each method used should be included (i.e. LCS data).

QA/QC Reports. These reports will include the general QC records, such as instrument calibration, routine monitoring of analytical performance, calibration verification, etc. Project-specific information from the QA/QC checks such as blanks (e.g., reagent, method), spikes (e.g., matrix, matrix spike duplicate, surrogate spike), calibration check samples (e.g., zero check, span check, and mid-range check), replicates, and so on should be included in these reports to facilitate data quality analysis.

Data Reporting Package Format and Documentation Control Report: The format of all data reporting packages must be consistent with the requirements and procedures used for data validation and data assessment described in Sections 8, 9, and 10 of the QAPP. The Project Manager will ensure that data are being recorded appropriately on the sample labels, sample tracking forms, and in the field notebook. All entries will be made using permanent ink, signed, and dated, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark that is signed and dated by the sampler. A similar data entry process will be followed by the contract laboratories. Only QC/Calibration summary forms will be provided at this time, unless analytical raw data is necessary.

Contract laboratories will be expected to provide a data package with the following components:

- Case Narrative:
- Date of issuance
- Laboratory analyses performed
- Any deviations from intended analytical strategy
- Laboratory batch number
- Numbers of samples and respective matrices
- Quality control procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples “as received”
- Discussion of whether or not sample holding times were met

- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the Laboratory QA Manager.

Chemistry Data Report:

- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory quality control checks
- Cross referencing of laboratory sample to project sample identification numbers
- Descriptions of data qualifiers
- Sample preparation and analyses for samples
- Sample and laboratory quality control results
- Results of (dated) initial and continuing calibration checks
- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results
- Results of tentatively identified compounds.

\*\* An electronic copy of the Analytical Data Report will be submitted in an MS Excel format containing the analytical test results \*\*

## 7. Special Training Requirements

No special training requirements are required for this project.

## 8. Quality Control Requirements

All analytical procedures are documented in writing as SOPs and each SOP includes QC information, which addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure. Examples of some of the QC samples that will be used during this project include:

- Method blanks
- Reagent/preparation blanks
- Instrument blanks
- Surrogate spikes
- Analytical spikes
- Field replicates
- Laboratory duplicates
- Matrix Spike/Matrix Spike Duplicate
- Laboratory control standards
- Internal standard areas for GC/MS or GC/ECD analysis; control limits.

The actual QC samples requirements will be dictated by the method requirements. Details on the use of each QC check are provided in the analytical SOPs provided for each measurement (see Appendix I). Method detection limits will be calculated for each analyte.

**Note:** Instrument calibration concentrations, method validation procedures, internal quality control protocols, analytical routines, maintenance and corrective actions, and the data reduction procedures are included in and will be performed as specified in the Standard Operation Procedures found in Appendix I and/or as required by the designated analytical methods.

## **8.1 Instrument/Equipment Testing, Inspection, and Maintenance Requirements**

The purpose of this section is to discuss the procedures used to verify that all instruments and equipment are maintained in sound operating condition, and are capable of operating at acceptable performance levels.

### *Testing, Inspection, and Maintenance*

The success of this project is dependent on well functioning field, analytical, and toxicological equipment. Preventative maintenance of this equipment is the key to reduce possible project delays due to faulty equipment.

As part of each laboratory's QA/QC program, a routine preventative maintenance program will be conducted to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance is carried out on a regular, scheduled basis and is documented in the laboratory instrument service logbook for each instrument.

## **8.2 Instrument Calibration and Frequency**

This section concerns the calibration procedures that will be used for instrumental analytical methods and other measurement methods that are used in environmental measurements. Calibration is defined as checking physical measurements against accepted standards.

### *Instrumentation Requiring Calibration*

All of the equipment used to analyze the sediment samples will require calibration, as will the water quality equipment used to monitor overlying water quality parameters in the sediment toxicity tests.

### *Calibration Methods That Will Be Used For Each Instrument*

Instrument calibration procedures are dependent on the method and corresponding SOP (see Appendix I). All ongoing calibration measurements must be within the requirements of the corresponding SOP to be considered adequate

### *Calibration Apparatus*

None of the analytical instruments will be calibrated using a calibration apparatus.

### *Calibration Standards*

The working linear range of an instrument should be established prior to performing sample analyses. Calibration standards as specified in the applicable methods and SOPs will be used when establishing the working linear range. The working linear range for a specific analysis should bracket the expected concentrations of the target analyte in the samples to be analyzed.

### *Calibration Frequency*

Instrument calibration is performed before sample analysis begins and is continued during sample analysis at the intervals specified within the applicable methods and SOPs (see Appendix I) in order to ensure that the data quality objectives are met. The verification of instrument stability is assessed by analyzing continuing calibration standards at regular intervals during the period that sample analyses are performed. Standards will be analyzed on a schedule as specified in the analytical SOPs. The concentration of the continuing calibration standard should be equivalent to the midpoint of the working linear range of the instrument.

Equipment logbooks will be maintained at each laboratory, in which will be recorded the usage, maintenance, calibration, and repair of instrumentation. These logbooks will be available during any audits that may be conducted.

*Thermometer Calibration*                      *See Appendix I*

*Deionized Water Supply*                      *See Appendix I*

*Analytical Balances*                              *See Appendix I*

Glassware Calibration/Verification      *See Appendix I*

## **8.3 Inspection/Acceptance Requirements for Supplies and Consumables**

The purpose of this section is to establish and document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of the project or task.

### *Identification of Critical Supplies and Consumables*

Critical supplies and consumables include sample bottles, gases, reagents, hoses, materials for decontamination activities, and distilled/deionized water. Each of the laboratories will utilize high quality supplies and consumables to reduce the chances of contaminating the samples. All water purification systems are tested on a regular basis to ensure that water produced is acceptable for use. Solvent blanks are run to verify the purity of solvents used in the organic analyses. The contract laboratories may also incorporate other measures, such as the dedicated use of glassware for certain analyses (e.g., inorganics, organics) or toxicity tests.

### *Establishing Acceptance Criteria*

Acceptance criteria must be consistent with overall project technical and quality criteria. Each of the laboratories should utilize their own acceptance criteria for normal operations with analyzing and/or testing contaminated sediments.

### *Inspection of Acceptance Testing Requirements and Procedures*

Each contract laboratory should document inspections of acceptance testing, including procedures to be followed, individuals responsible, and frequency of evaluation. In addition, handling and storage conditions for supplies and consumables should be documented.

### *Tracking and Quality Verification of Supplies and Consumables*

Procedures should be established to ensure that inspections or acceptance testing of supplies and consumables are adequately documented by permanent, dated, and signed records or logs that uniquely identify the critical supplies or consumables, the date received, the date tested, the date to be retested (if applicable), and the expiration date. These records should be kept by the responsible individual(s) at each laboratory. In order to track supplies and consumables, labels with the information on receipt and testing should be used. These or similar procedures should be established to enable project personnel to: 1) verify, prior to use, that critical supplies and consumables meet the project objectives; and 2) ensure that supplies and consumables that have not been tested, have expired, or do not meet acceptance criteria are not used for the project.

## **8.4 Data Management**

This section will present an overview of all mathematical operations and analyses performed on raw data to change their form of expression, location, quantity, or dimensionality. These operations include data recording, validation, transformation, transmittal, reduction, analysis, management, storage, and retrieval.

### *Laboratory Data Recording*

All raw analytical and toxicity data will be recorded in numerically identified laboratory notebooks or data sheets. The data will be promptly recorded in black ink on appropriate forms that are initialed and dated by the person collecting the data. Changes to recorded data are made in black ink, with a single line cross-out, initials, and date. No "whiteout" will be allowed.

If a laboratory has the capability to directly enter or download the data into a computerized data logger, then this is preferable. All labs shall download data directly into a computerized database. Sample data are recorded along with other pertinent information, such as the sample identification number. Other details which will also be recorded include: the analytical method used (SOP #), name of analyst, the date of analysis or toxicity test, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook or data sheet will be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Laboratory Supervisors will take place prior to final data reporting. Records of notebook entry inspections are maintained by the Laboratory QA Officer.

### *Data Verification*

The method, instrument, or system should generate data in a consistent, reliable, and accurate manner. Data validation will be shown by meeting acceptable QC limits for analytical parameters and sediment toxicity tests. In addition, the application of preventative maintenance activities and internal QA/QC auditing will ensure that field and laboratory generated data will be valid. Quality control data (e.g., laboratory duplicates, matrix spikes, matrix spike duplicates, and performance of negative controls) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data verification is performed by a second designated senior/experienced staff at the technical level where QC results, hold times, and instrument calibration is evaluated. All QA requirements are programmed into automated systems and flagged where appropriate.

### *Data Transformation*

Data transformations result from calculations based on instrument output, readings, or responses. The procedures for converting calibration readings into an equation that will be applied to measurement readings are given in the SOPs for analytical parameters (Appendix I).

### *Data Transmittal*

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another. Some examples of data transmittal are copying raw data from a notebook onto a data entry form for keying into a computer file and electronic transfer of data over a computer network. The transmittal of field data will be double-checked by the PI. The transmittal of laboratory data will be checked by the individual analyst with periodic checks by the Laboratory Project Manager and/or QA Officer.

### *Data Reduction*

Data reduction includes all processes that change the number of data items. Each laboratory has their own data reduction techniques, as is usually documented in their QA Manual. For the analytical results, data reduction will involve calculating the arithmetic mean and standard deviation of field and laboratory replicates.

### *Data Analysis*

Data analysis will involve comparing the surficial contaminant concentrations to qualitative values contained in Table 1. The analysis shall be performed by the USEPA Project Manager.

### *Data Tracking*

Data management includes tracking the status of data as they are collected, transmitted, and processed. Each laboratory will have its own data tracking system in place.

### *Data Storage and Retrieval*

Each contract laboratory will have its own data storage and retrieval protocols. USEPA-GLNPO will retain all the analytical data packages in the project files for this study. In addition, the sediment contaminant data will be added to GLNPO's contaminated sediment database.

## **8.5 Data Acquisition Requirements (Non-Direct)**

We will be utilizing historical sediment chemistry data and fish tissue data for this project. Prior to utilizing this data, the USEPA Project Manager will be responsible for verifying the quality of this data. Historical data will only be used if original laboratory reports are available for review

and assessment. At a minimum, the USEPA Project Manager will utilize the checklists contained in Appendix J to verify the quality of the historical data. If the historical data does not meet the requirements of the checklist, or if the Project Manager is unable to ascertain the quality of the historical data, this data will not be used in the project analysis.

Additionally, sets of screening values will be used to evaluate the potential impacts of the contaminant concentrations found in the sediments during this survey. All parameter data will be compared to existing sediment quality guidelines available in *MacDonald et. al.* (2000) and *Persuad et. al.* (1993). All of these screening levels were specifically developed for freshwater ecosystems and have been published in peer reviewed journals and documents. Therefore, these guidelines are considered sufficient for a screening level analysis of sediment data.

Water surface elevation data will be obtained from the NOAA web page. Only data from the "verified/historical water level data" page will be utilized in the study. However, NOAA has attached the following disclaimer on data from this web page:

**"These raw data have not been subjected to the National Ocean Service's quality control or quality assurance procedures and do not meet the criteria and standards of official National Ocean Service data. They are released for limited public use as preliminary data to be used only with appropriate caution."**

Since the water surface elevation data is non-critical data, this preliminary data is sufficient for our needs.

## **9. Assessment and Oversight**

### **9.1 Assessment and Response Actions**

During the planning process, many options for sampling design, sample handling, sample cleanup and analysis, and data reduction are evaluated and chosen for the project. In order to ensure that the data collection is conducted as planned, a process of evaluation and validation is necessary. This section of the QAPP describes the internal and external checks necessary to ensure that:

- All elements of the QAPP are correctly implemented as prescribed.
- The quality of the data generated by implementation of the QAPP is adequate.
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

The most important part of this section is documenting all planned internal assessments. Generally, internal assessments are initiated or performed by the QA Officer.

### *Assessment of Subsidiary Organizations*

Two types of assessments of the subsidiary organizations can be performed as described below.

- *Management Systems Review (MSR)*. A form of management assessment, this process is a qualitative assessment of a data collection operation or organization to establish whether the prevailing quality management structure, policies, practices, and procedures are adequate for ensuring that the type and quality of data needed are obtained. The MSR is used to ensure that sufficient management controls are in place and carried out by the organization to adequately plan, implement, and assess the results of the project.
- *Readiness Reviews*. A readiness review is a technical check to determine if all components of the project are in place so that work can commence on a specific phase.

It is anticipated that a readiness review by each contract laboratory project manager will be sufficient for this project. No management systems review is anticipated for this project. A pre-project QA/QC conference call (already held) and submittal of laboratory certifications and/or QA plans shall suffice as a MSR.

### *Assessment of Project Activities*

Assessment of project activities can involve the following tasks:

- Surveillance
- Technical Systems Audit (TSA)
- Performance Evaluation (PE)
- Audit of Data Quality (ADQ)
- Peer Review
- Data Quality Assessment.

Surveillance will be the primary assessment technique of project activities. This will most readily occur by the Project Manager and QA Officer of each contract laboratory.

### *Number, Frequency, and Types of Assessments*

Due to the short-term nature of this project for the contract laboratories, no types of assessments are planned other than general surveillance, a data quality assessment by USACE representatives, and peer review by USACE and USEPA.

### *Assessment Personnel*

Internal audits of the contract laboratories are regularly performed by their respective QA Officers.

### *Schedule of Assessment Activities*

External audits by the GLNPO QA Officer and/or the GLNPO Project Manager is up to his/her discretion. The scheduling of regular internal audits at contract labs is at the discretion of the respective QA Officers.

### *Reporting and Resolution of Issues*

Any audits or other assessments that reveal findings of practice or procedure that do not conform to the written QAPP need to be corrected as soon as possible. The Laboratory Project Manager and Laboratory QA Officer need to be informed immediately of critical deviations that compromise the acceptability of the test. For any critical deviations from the QAPP (i.e., elevated detection levels, surrogate recoveries outside control limits, etc.) that cannot be corrected within the laboratories standard procedure, the Laboratory Project Manager must contact both the USEPA Project Manager and the USACE Project Coordinator within 24-hours of being informed of the deviation. The laboratory project manager should be ready to provide suggestions for corrective action. For non-critical deviations, they need to be informed by the next business day.

Corrective actions should only be implemented after approval by both the USACE and the USEPA Project Managers. If immediate corrective action is required, approvals secured by telephone from the USEPA Project Manager should be documented in an additional memorandum. In general communications from the laboratories should follow the chain-of-command as shown in Figure 1. However, if the subcontract laboratories are unable to contact the Lakeshore Engineering Services Project Manager on any time-critical matter, the laboratories shall contact either the USACE Project Coordinator or USEPA Project Manager as necessary.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem will be responsible for notifying the project manager. Implementation of corrective actions will be confirmed in writing through the same channels. Each laboratory shall issue a nonconformance report for each nonconformance condition.

Corrective actions in the laboratory may occur prior to, during, and after initial analysis. A number of conditions, such as broken sample containers, multiple phases, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with laboratory analysts and section leaders, it may be necessary for the Laboratory QA Officer to approve the implementation of corrective actions. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective actions of samples, including additional sample extract cleanup and automatic re-injection/reanalysis when certain quality control criteria are not met.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- Blanks contain target analytes above acceptable levels
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- QC limits for sediment toxicity tests are not met
- Deficiencies are detected by the Laboratory and/or GLNPO QA Officer(s) during any internal or external audits or from the results of performance evaluation samples
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, experimental set-up, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Project Manager and/or Laboratory QA Officer for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the Laboratory QA Officer.

These corrective actions are performed prior to release of the data from the laboratory. The corrective actions will be documented in both the laboratories corrective action log and the narrative data report sent from the laboratory to the Lakeshore Engineering Services Project Manager.

If corrective action does not rectify the situation, the laboratory will contact Lakeshore Engineering Services Project Manager who will then contact the USACE Project Coordinator and USEPA Project Manager to discuss details of the corrective actions and required future actions.

## **9.2 Reports to Management**

### *Responsible Organizations*

Written QC data and appropriate QA/QC reports generated by the laboratories shall be included in the Analytical Data Report. The Analytical Data Report will be provided by the laboratories to the Project Manager by the persons identified in Section 1.3 whenever sample measurements are reported. The QC section of the Analytical Data Report should include the QC data (including results, recoveries, and RPDs), any non-conformance reports, and chains of custody. The report should give detailed results of analysis of QC samples, and provide information on the precision, accuracy, and completeness for each sample run. These written reports will note any significant QA/QC problems encountered during sample analyses, as well as state the corrective actions taken.

Any serious QA problems needing immediate decisions will be discussed orally between the USACE Project Coordinator and laboratory staff, with such discussions denoted in writing. Communication should follow the chain-of-command summarized in Figure 1. These problems will be noted in the final project report to the USEPA Project Coordinator.

The USACE-Project Coordinator will provide summary QA/QC information in the final written report to USEPA. This report will include information on adherence of measurements to the QA objectives. The final report will contain detailed discussions of QA/QC issues, including any changes in the QAPP, a summary of the contract laboratories QA/QC reports, results of any internal performance audits, any significant QA/QC problems, detailed information on how well the QA objectives were met, and their ultimate impact on decision making. The following is a list of items to be included in the final project report:

- Changes in the QAPP
- Results of any internal system audits
- Significant QA/QC problems, recommended solutions, and results of corrective actions

- Data quality assessment in terms of precision, accuracy, representativeness, completeness, and sensitivity
- Indication of fulfillment of QA objectives
- Limitations on the use of the measurement data.

## 10. Data Validation and Usability

The USEPA Project Manager will make a final decision regarding the validity and usability of the data collected during this project. The project manager will evaluate the entire sample collection, analysis, and data reporting processes to determine if the data is of sufficient quality to meet project objectives. Data validation involves all procedures used to accept or reject data after collection and prior to use. These include screening, editing, verifying, and reviewing through external performance evaluation audits. Data validation procedures ensure that objectives for data precision and bias will be met, that data will be generated in accordance with the QA project plan and SOPs, and that data are traceable and defensible. The process is both qualitative and quantitative and is used to evaluate the project as a whole.

### *Procedures Used to Validate Field Data*

Procedures to evaluate field data for this project primarily include checking for transcription errors and reviewing field notebooks. This task will be the responsibility of the project manager.

### *Procedures Used to Validate Laboratory Data*

The respective Laboratory QA Officer will conduct a systematic review of the analytical data for compliance with the established QC criteria based on the spike, duplicate, and blank results provided by the laboratory. All technical holding times will be reviewed, the laboratory analytical instrument performance will be evaluated, and results of initial and continuing calibration will be reviewed and evaluated.

Upon receipt of the draft laboratory report, the U.S. Army Corps of Engineers will perform a thorough QA/QC review of the chemical data. At a minimum, this review will include an analysis of:

- Sample Receipt Verification/Documentation
- Detection Limits
- Surrogate Recoveries
- Laboratory QC Documentation and Results
- Holding Time Data
- Process Bias and Sensitivity
- MS/MSD Recoveries
- Analytical Method Documentation

At the conclusion of the review, the U.S. Army Corps of Engineers will prepare a report describing the results of the review, providing recommendations on data items requiring corrective action or further documentation/information, and drawing conclusions as to the usability of the data provided. A draft report will be provided to both the analyzing laboratories and the U.S. EPA project manager for review and comment prior to finalizing conclusions and recommendations.

The data review will identify any out-of-control data points and data omissions, and the Laboratory QA Officer will interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analysis may be made by the USEPA Project Manager based on the extent of the deficiencies and their importance in the overall context of the project.

Additionally, the USEPA project manager will compare all field and laboratory duplicates for RPD. Based on the results of these comparisons, the USEPA project manager will determine the acceptability of the data. One hundred percent of the analytical and toxicity data will be validated. Reconciliation of laboratory and field duplicates shall be the responsibility of the USEPA project manager.

Finally, the USEPA project manager will compare the laboratory methods and results to the QA/QC Review checklists contained in Appendix J. Separate checklists are for chemistry data and toxicity data. Any critical problems identified by these checklists that we are unable to rectify through corrective actions, may be cause for rejecting portions or all of the data provided.

## 11. References

MacDonald, Donald D., Ingersoll, C.G., and Berger, T.A., 2000, "Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems", *Archives of Environmental Contamination and Toxicology*, 39:20-31

NATO, 1988. International Toxicity Equivalency Factor (I-tef) Method of Risk Assessment for Complex Mixtures of Dioxins and Related Compounds. North Atlantic Treaty Organization. Report Number 176.

Persuad, D., Jaaguamagi, R., and Hayton, A. (1993), "Guidelines for the Protection and Management of Aquatic Sediment in Ontario", Water Resources Branch, Ontario Ministry of the Environment, Toronto, Ontario.

USEPA (2000), *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates*, Office of Research and Development, U.S. Environmental Protection Agency, Washington, DC, EPA/600/R-99/064.